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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6 :
A61K 7/50, 7/06, 7/00

A1

(11) International Publication Number: **WO 99/32079**

(43) International Publication Date: 1 July 1999 (01.07.99)

(21) International Application Number: PCT/EP98/08150

(22) International Filing Date: 10 December 1998 (10.12.98)

(30) Priority Data:
9726970.8 19 December 1997 (19.12.97) GB

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(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published
With international search report.
Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.

(54) Title: SHAMPOO COMPOSITIONS

(57) Abstract

An optically–clear shampoo composition comprising: (a) microemulsified particles of a high viscosity silicone having a particle size of ≤ 0.15 microns; (b) at least one surfactant; (c) a deposition polymer which is a cationic derivative of guar gum having a molecular weight in the range of 50,000 to 200,000, a nitrogen content of from 1.5 to 13.8% by weight nitrogen based on the weight of the polymer molecule, a degree of cationization of from 0.15 to 3.0, and (d) an aqueous carrier.

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SHAMPOO COMPOSITIONS

FIELD OF THE INVENTION

The present invention relates to shampoo compositions. More particularly, the invention relates to hair shampoo compositions which include a silicone microemulsion and a deposition polymer.

BACKGROUND AND PRIOR ART

The use of silicones in hair shampoos is well known. Generally, dispersed droplets of the silicone oil are suspended in the composition, which is then applied to the hair to deposit the silicone material on the hair shaft.

It is known in the art that oily cosmetic agents such as silicones can be incorporated into cosmetic compositions by means of microemulsification, whereby the silicone is present as stably emulsified droplets of a particle size of the order of 0.15 microns or less.

In EP A 0 529 883 there is disclosed a hair shampoo comprising a silicone microemulsion in combination with a cationic deposition polymer. The viscosity of the silicone microemulsion used is 15 000 centistokes. This shampoo gives satisfactory deposition of the microemulsion onto hair, but the conditioning benefit is not sufficient for many people.

EP A 0 674 898 proposes an improvement to this, and discloses that shampoo compositions with good mechanical

stability, high optical transparency or translucency and excellent conditioning ability can be obtained by utilising a high viscosity microemulsified silicone oil in combination with a cationic deposition polymer. The preferred, and exemplified , deposition polymer is a cationic cellulose ether derivative. This is said to give good clarity and adequate flocculation on dilution during use. Cationic guar gum derivatives, in contrast, are said to be unsuitable for mild shampoo formulations because they give poor clarity.

A problem with the high viscosity microemulsion/cationic cellulose ether type formulations as disclosed in EP A 0 674 898 is that they tend to deliver a heavy or slimy feel to the hair during the wet stage of treatment.

We have surprisingly found that this problem can be overcome by utilising a specific type of cationic guar gum derivative in conjunction with a high viscosity microemulsion.

Shampoo formulations according to our invention have been found to give excellent translucency/transparency, conditioning ability and improved wet sensory feel.

SUMMARY OF THE INVENTION

The present invention provides an optically-clear shampoo composition comprising :

(a) microemulsified particles of a high viscosity silicone having a particle size of ≤ 0.15 microns;

(b) at least one surfactant;

(c) a deposition polymer which is a cationic derivative of guar gum having a molecular weight in the range of 50,000 to 200,000, a nitrogen content of from 1.5 to 13.8% by weight nitrogen based on the weight of the polymer molecule, a degree of cationization of from 0.15 to 3.0, and

(d) an aqueous carrier.

DETAILED DESCRIPTION AND PREFERRED EMBODIMENTS

Optical Clarity

For the purposes of the present invention, the term "optically clear" is used to define a composition that is transparent (transmitting light without distortion). Which means that the size of the particles in the composition are reduced to a size where they are not observable with optical (visual) means. According to the present invention, "optically clear" is further defined by NTUs (Nephelometric Turbidity Units), which is the unit of measure for the turbidity or haze of a liquid. NTUs range from 0.04 to 1,000 or higher.

The turbidity of liquid samples may be tested by light scattering according to Corporate Test Method (CTM) 0851, referenced in EP 0 514 934 (Dow Corning). Turbidity or haze of liquids caused by the presence of suspended particulate matter is measured by light scattering. Light is passed through a flat bottom cell containing the liquid. As the light beam strikes particles in the liquid, some of the light is scattered at right angles to the incident beam and is received by a photomultiplier tube. The photomultiplier

tube converts the light energy into an electrical signal which is measured on a meter. The unit of measure is the NTU and is based on formalin suspensions as standards. The range of turbidities detectable is 0.04 to 1,000 NTUs. The haze value of a relatively turbid solution is about 100 NTUs or higher and mixtures with a slight haze give values of 20 to 50 NTUs.

In contrast, the compositions of the present invention have an average haze value of 3 to 10 NTUs.

High Viscosity Silicone

The shampoo composition of the invention comprises microemulsified particles of a high viscosity silicone having a particle size of < 0.15 microns.

Preferably the microemulsified particles will have a particle size of from 0.01 to 0.15 microns.

Particle size may be measured by means of a laser light scattering technique, using a 2600D Particle Sizer from Malvern Instruments.

As used herein, the term high viscosity means in excess of 30 000 centistokes. The viscosity preferably exceeds 50 000 centistokes. The viscosity being measured is the viscosity of the silicone itself and not that of the emulsion or the final shampoo composition. The viscosity can be measured by means of a glass capillary viscometer as set out further in Dow Corning Corporate Test Method CTM004 July 20 1970.

Suitable silicones may be one or more polyalkyl siloxanes, one or more polyalkylaryl siloxanes, or mixtures thereof. The silicone is insoluble in the aqueous matrix of the composition and so is present as dispersed particles, in a microemulsified form.

Suitable polyalkyl siloxanes include polydimethyl siloxanes which have the CTFA designation dimethicone, having a viscosity of up to 100,000 centistokes at 25 degrees C. These siloxanes are available commercially from the General Electric Company as the Viscasil series and from Dow Corning as the DC 200 series. Also suitable is polydiethyl siloxane.

Also suitable are silicone gums, such as those described in US Pat. No. 4,152,416 (Spitzer), and on General Electric Silicone Rubber product Data Sheet SE 30, SE 33, SE 54 and SE 76. "Silicone gum" denotes polydiorganosiloxanes having a molecular weight of from 200,000 to 1,000,000 and specific examples include polydimethyl siloxane polymers, polydimethyl siloxane/diphenyl/methylvinylsiloxane copolymers, polydimethylsiloxane/methylvinylsiloxane copolymers and mixtures thereof.

Aminofunctional silicones which have the CTFA designation amodimethicone, are also suitable for use in the compositions of the invention, as are polydimethyl siloxanes having hydroxyl end groups (which have the CTFA designation dimethiconol).

Various methods of making microemulsions of particles of silicones for use in the invention are available and are well known and documented in the art.

One particularly preferred technique for making silicone microemulsions is that described in EP-A-228575.

In that document there is described a method of making a stable microemulsion of high molecular weight silicone polymer and water by sequentially adding at an effective rate a standard emulsion comprising polydiorganosiloxane precursor, surfactant and water to a polymerization catalyst medium while mixing to form a clear, stable aqueous microemulsion of polydiorganosiloxane.

Another method of making suitable microemulsions for use in the invention are described in EP-A-0 138 192.

Alternatively, suitable microemulsions for use in the invention are commercially available in a pre-microemulsified form. This is particularly preferred since the pre-formed microemulsion can be incorporated into the final shampoo composition by simple mixing. An example of a suitable pre-formed microemulsion is microemulsion DC2-1870, available from Dow Corning. This is a microemulsion of dimethiconol of viscosity 60,000 centistokes.

The amount of silicone incorporated into the compositions of the invention depends on the type of composition and the material used. A preferred amount is from 0.01 to about 10% by weight although these limits are not absolute. The lower limit is determined by the minimum level to achieve conditioning and the upper limit by the maximum level to avoid making the hair and/or skin unacceptably greasy. We have found that an amount of from 0.5 to 1.5%, e.g. about 0.8% by weight of the total shampoo composition, is a

particularly suitable level of silicone in shampoo compositions of the invention.

Surfactant

Shampoo compositions of the invention comprise one or more surfactants, at least as emulsifying agents for the microemulsifed particles of high viscosity silicone.

Suitable emulsifying agents are well known in the art and include anionic and nonionic surfactants. Examples of anionic surfactants used as emulsifying agents for the high viscosity silicone are alkylarylsulphonates, e.g., sodium dodecylbenzene sulphonate, alkyl sulphates e.g., sodium, lauryl sulphate, alkyl ether sulphates, e.g., sodium lauryl ether sulphate nEO, where n is from 1 to 20 alkylphenol ether sulphates, e.g., octylphenol ether sulphate nEO where n is from 1 to 20, and sulphosuccinates, e.g., sodium dioctylsulphosuccinate.

Examples of nonionic surfactants used as emulsifiying agents for the high viscosity silicone are alkylphenol ethoxylates, e.g., nonylphenol ethoxylate nEO, where n is from 1 to 50, alcohol ethoxylates, e.g., lauryl alcohol nEO, where n is from 1 to 50, ester ethoxylates, e.g., polyoxyethylene monostearate where the number of oxyethylene units is from 1 to 30.

Further surfactants may be present as an additional ingredient if sufficient for cleansing purposes is not provided as the emulsifier for the microemulsified particles of high viscosity silicone. It is preferred that shampoo compositions of the invention comprise at least one further

surfactant (in addition to that used as emulsifying agent for the high viscosity silicone), to provide a cleansing benefit. The cleansing surfactant is typically selected from anionic, nonionic, amphoteric and zwitterionic surfactants, and mixtures thereof. The cleansing surfactant may be the same surfactant as the emulsifier, or may be different.

Suitable anionic cleansing surfactants for shampoo compositions of the invention include the alkyl sulphates, alkyl ether sulphates, alkaryl sulphonates, alkanoyl isethionates, alkyl succinates, alkyl sulphosuccinates, N-alkoyl sarcosinates, alkyl phosphates, alkyl ether phosphates, alkyl ether carboxylates, alpha-olefin sulphonates and acyl methyl taurates, especially their sodium, magnesium ammonium and mono-, di- and triethanolamine salts. The alkyl and acyl groups generally contain from 8 to 18 carbon atoms and may be unsaturated. The alkyl ether sulphates, alkyl ether phosphates and alkyl ether carboxylates may contain from one to 10 ethylene oxide or propylene oxide units per molecule, and preferably contain 2 to 3 ethylene oxide units per molecule.

A particularly preferred class of anionics are the acyl methyl taurates, since these confer exceptional mildness to the shampoo compositions of the invention. Illustrative of this class are the sodium salts of C_{12}-C_{22} fatty acid amides of N-methyl taurine. Examples are sodium methyl cocoyl taurate, sodium methyl lauroyl taurate, sodium methyl stearoyl taurate and sodium methyl palmitoyl taurate.

Acylated collagen polypeptide is another specifically preferred class of surfactant, an example being cocoyl hydrolysed collagen.

Nonionic cleansing surfactants suitable for use in shampoo compositions of the invention may include condensation products of aliphatic (C_8-C_{18}) primary or secondary linear or branched chain alcohols or phenols with alkylene oxides, usually ethylene oxide and generally having from 6 to 30 ethylene oxide groups. Other suitable nonionics include alkylpolyglycosides and mono- or di-alkyl alkanolamides. Examples of the latter nonionics include coco mono- or di-ethanolamide and coco mono-isopropanolamide.

Amphoteric and zwitterionic cleansing surfactants suitable for use in compositions of the invention may include alkyl amine oxides, alkyl betaines, alkyl amidopropyl betaines, alkyl sulphobetaines (sultaines), alkyl glycinates, alkyl carboxyglycinates, alkyl amphopropionates, alkylamphoglycinates and alkyl amidopropyl hydroxysultaines. Examples include lauryl amine oxide, cocodimethyl sulphopropyl betaine and preferably lauryl betaine, cocamidopropyl betaine and sodium cocamphopropionate.

The total amount of surfactant (including that used as emulsifier for the microemulsified particles of high viscosity silicone) in shampoo compositions of the invention is generally from 0.1 to 50% by weight, preferably from 5 to 30%, more preferably from 10% to 25% by weight of the total shampoo composition.

We have found that exceptional optical clarity and stability, across a wide range of temperatures, can be achieved with the following combination of cleansing surfactants in the stated amounts:

from 4 to 8% by weight of the total shampoo composition of acyl methyl taurate and/or acylated collagen polypeptide; and up to 8% by weight of the total shampoo composition of cocamidopropylbetaine.

Deposition polymer

Shampoo compositions of the invention contain a cationic deposition polymer which is a cationic derivative of guar gum.

By "deposition polymer" is meant an agent which enhances deposition of the particles of silicone from the shampoo composition of the invention onto the intended site during use, i.e. the hair and/or the scalp.

Guar gum occurs naturally as the principal component of the seed of the guar plant, *cyamopsis tetragonalobus*. The guar molecule is essentially a straight chain mannan branched at quite regular intervals with single membered galactose units on alternative mannose units. The cationic derivatives are obtained by reaction between the hydroxyl groups of the polygalactomannan and reactive quaternary ammonium groups.

Cationic guar gum derivatives in general are well known and widely utilised in shampoos. Examples are those hydroxypropyltrimonium chloride derivatives of guar sold by Rhone-Poulenc under their JAGUAR trademark series, of which a particlularly preferred type is JAGUAR C-13-S.

We have found, however, that JAGUAR C-13-S is not a suitable type of cationic guar gum derivative for shampoos of the

present invention, since it tends to adversely affect the optical clarity of the formulation.

Instead, we have found that a specific type of cationic guar gum derivative is necessary in order to achieve the excellent translucency/transparency, conditioning ability and improved wet sensory feel observed with shampoos of the invention.

Cationic guar gum derivatives suitable for use in the present invention must fulfill specific criteria in relation to the parameters of molecular weight, nitrogen content and degree of cationization:

Molecular Weight

In cationic guar gum derivatives suitable for use in the present invention, the molecular weight ranges from 50,000 to 200,000, preferably from 75,000 to 150,000. For optimum performance, the molecular weight of the cationic guar gum derivative is ideally about 100,000.

Nitrogen Content

The nitrogen content can be measured by the Kjehldahl method. In cationic guar gum derivatives suitable for use in the present invention, the nitrogen content ranges from 1.5 to 13.8% , preferably from 1.8 to 3.2% by weight nitrogen based on the weight of the polymer molecule. For optimum performance, the nitrogen content ideally ranges from 2.2 to 2.8% by weight nitrogen based on the weight of the polymer molecule.

Degree of Cationization

By "degree of cationization" is meant the degree of substitution of the polymer molecule with cationic groups. This is calculated using the following formula:

Degree of cationization = nitrogen content (i.e wt% based on the weight of the polymer molecule as described above)/ {atomic weight of nitrogen(14)/ [average molecular weight for the monosaccharide unit in the cationic guar gum + average molecular weight for the cationic substituent on the cationic guar gum]} x 100

So, for example, for a cationic guar gum in which (i) the monosaccharide units are D-galactose and D-mannose in a 1:2 ratio; (ii) the nitrogen content is 2.5 wt% based on the weight of the polymer molecule; (iii) the cationic substituent is hydroxypropyltrimethylammonium chloride, application of the above formula gives a degree of cationization of 0.56.

In cationic guar gum derivatives suitable for use in the present invention, the degree of cationization ranges from 0.15 to 3.0, preferably from 0.25 to 0.65. For optimum performance, the degree of cationization ideally ranges from 0.33 to 0.57.

We have found that cationic deposition polymers which are cationic derivatives of guar gum fulfilling the above specific criteria give excellent optical clarity to the formulation, in conjunction with the appropriate amount of water-soluble simple salt.

A suitable cationic derivative of guar gum for use in the shampoo composition of the invention is Rhaballgum CG-M 8M, available commercially from Dainihon Pharmaceutical.

The deposition polymer may be present in an amount of from about 0.01 to about 10% by weight of the total shampoo composition, preferably from about 0.01 to about 1% by weight, even more preferably from about 0.04 to about 0.5% by weight. A particularly preferred level is about 0.3% by weight, since this delivers excellent wet sensory properties as well as optical clarity.

Salt level

The total level of salt in shampoo compositions of the invention has been found to be important for obtaining good optical properties across a wide temperature range.

The total salt level in the shampoo composition may be adjusted, for example, by the addition of a simple salt. By a simple salt is meant a salt which dissolves in water and ionizes but whose ions do not aggregate in solution as, for example, do the ions of a surface active agent which aggregate to form micelles. Suitable salts include the water-soluble alkali metal, alkaline earth metal and ammonium salts (including substituted ammonium salts) of inorganic acids and organic acids containing a carbon chain of not more than about 6 carbon atoms. Chlorides, nitrates and sulphates are preferred inorganic salts and suitable salts of organic acids include the acetates, benzoates and citrates. The molecular weight of the salt will usually be less than 600. Specific examples of suitable salts are sodium chloride and sodium benzoate.

The level of salt required will depend on the precise nature of the salt used and the particular surfactant system employed.

For a shampoo based on an acyl methyl taurate surfactant system, as is preferred for shampoos of the present invention, we have found that a total sodium chloride level of from about 1 to 2.5%, optimally from 1.18 to 2.34%, by weight based on total weight of the shampoo composition gives excellent optical clarity, even in very cold conditions.

Aqueous carrier

The shampoo compositions of the invention comprise an aqueous carrier, water forming the basis of the continuous phase of the microemulsion. The compositions preferably comprise water in an amount of from about 20 to about 99% by weight of the total shampoo composition.

Product Form

The compositions of the invention are preferably rinse-off compositions, i.e., suitable for applying to the hair, left thereon for an appropriate period of time and then rinsed off with water.

Compositions of the invention suitably have a viscosity of from 1000-2000 cps(Brookfield viscometer, LVT type, Rotor No.3, 12 rpm, after 30 sec. at 25°C).

Typically such viscosities are achieved by the addition of an appropriate thickener. Suitable thickeners include esters

of fatty acids and polyethylene glycols such as for example the di-ester of stearic acid and a polyethylene glycol of molecular weight between 2,000 and 15,000, preferably between 5,000 and 10,000, optimally between 7,300 and 9,500.

Optional Ingredients

Depending upon the type of shampoo employed, one or more additional ingredients conventionally incorporated into shampoo formulations may be included in the compositions of the invention. Such additional ingredients include antibacterial agents, antidandruff agents, foam boosters, perfumes, colouring agents, preservatives, proteins, polymers, buffering or pH adjusting agents, moisturising agents, herb or other plant extracts and other natural ingredients.

The invention is further illustrated by way of the following non-limiting Example:

EXAMPLE

Three formulations were made up having the ingredients shown in the following Table.

Formula A and Formula B are comparative examples. Formula C is an example according to the present invention.

The three formulations were evaluated for wet and dry conditioning performance and assessed visually for optical clarity. The results are shown at the foot of the Table, and demonstrate how Formula C (the example of the invention) outperforms Formula A and Formula B (the comparative

examples) in showing good performance across all three attributes.

Ingredients	Formula A (w/w %)	Formula B (w/w %)	Formula C (w/w %)
Anionic surfactant	10	8	8
Amphoteric surfactant	5	8	8
Polymer JR400 (1)	0.2		
Jaguar C-13-S (2)		0.3	
Rhaballgum CG-M 8M (3)			0.3
Silicone (4)	1	1	1
Preservatives	0.4	0.6	0.6
Perfume	0.4	0.4	0.4
Aqua	balance	balance	balance
conditioning at wet stage	bad	good	good
conditioning at dry stage	bad	good	good
clarity of formulation	good	bad	good

(1) Quaternary ammonium derivative of cellulose ether, ex Union Carbide

(2) Guar hydroxypropyltrimonium chloride, ex Rhone-Poulenc

(3) Guar hydroxypropyltrimonium chloride, ex Dainihon Pharmaceutical

(4) 60,000 centistokes dimethiconol, added as microemulsion DC2-1870 (25% active ingredient) ex Dow Corning.

CLAIMS

1. An optically-clear shampoo composition comprising :

(a) microemulsified particles of a high viscosity silicone having a particle size of ≤ 0.15 microns;

(b) at least one surfactant;

(c) a deposition polymer which is a cationic derivative of guar gum having a molecular weight in the range of 50,000 to 200,000, a nitrogen content of from 1.5 to 13.8% by weight nitrogen based on the weight of the polymer molecule, a degree of cationization of from 0.15 to 3.0, and

(d) an aqueous carrier.

2. A shampoo composition according to claim 1, in which the high viscosity silicone is pre-microemulsified dimethiconol of viscosity greater than 50,000 centistokes.

3. A shampoo composition according to claim 1 or claim 2, in which the at least one surfactant comprises an acyl methyl taurate.

4. A shampoo composition according to any preceding claim, in which the deposition polymer is a cationic derivative of guar gum having a molecular weight of about 100,000, a degree of cationization of from 0.33 to 0.57 and a nitrogen content of from 2.2 to 2.8% by weight nitrogen based on the weight of the polymer molecule.

5. An optically-clear shampoo composition comprising:

(a) from 0.5 to 1.5% by weight of the total shampoo composition of pre-microemulsified particles of dimethiconol having a particle size of ≤ 0.15 microns and a viscosity greater then 50,000 centistokes;

(b) from 4 to 8% by weight of the total shampoo composition of acyl methyl taurate surfactant and/or acylated collagen polypeptide surfactant and up to 8% by weight of the total shampoo composition of cocamidopropylbetaine surfactant;

(c) from 0.04 to 0.5% by weight of the total shampoo composition of a deposition polymer which is a cationic derivative of guar gum having a molecular weight of about 100,000, a degree of cationization of from 0.33 to 0.57 and a nitrogen content of from 2.2 to 2.8% by weight nitrogen based on the weight of the polymer molecule;

(d) a total sodium chloride level of from 1 to 2.5% by weight based on total weight of the shampoo composition;

(e) a thickener which is a di-ester of stearic acid and a polyethylene glycol of molecular weight between 2,000 and 15,000, and

(f) an aqueous carrier.

INTERNATIONAL SEARCH REPORT

Inte. .onal Application No
PCT/EP 98/08150

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 A61K7/50 A61K7/06 A61K7/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 529 883 A (UNILEVER PLC ;UNILEVER NV (NL)) 3 March 1993 cited in the application see claims 1-13	1,4
X	EP 0 674 898 A (UNILEVER PLC ;UNILEVER NV (NL)) 4 October 1995 cited in the application see page 4, line 5 - line 14; claims 1-10	1,4
X	WO 95 09599 A (UNILEVER PLC ;UNILEVER NV (NL)) 13 April 1995 see page 11, line 28 - page 13, line 24 see claims 1-10	1,2,4
A	WO 97 22329 A (UNILEVER PLC ;UNILEVER NV (NL)) 26 June 1997 see claims 1-8; example 6	

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
1 June 1999	08/06/1999

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